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Impact of Partially Molten Plasma-Sprayed Zirconia Particles on Glass Surfaces

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Abstract

Plasma-sprayed yttria-stabilized zirconia particles (~40 μm diameter) were photographed during impact (velocity ~200 m/s) on a glass surface that was maintained at either room temperature or 400°C. A droplet that approached the surface was sensed using a photodetector and after a known delay, a light source was triggered to illuminate the particle in order to photograph it with a CCD camera. A rapid two-color pyrometer was used to collect the thermal radiation from the particles to follow the evolution of their temperature and size, in-flight and after impact. The fully molten particles spread into a thin film liquid splat after impacting the surfaces. The partially molten particles disintegrated into small satellite fragments immediately upon impact. The surface area, as indicated by the pyrometric signals, of the partially molten particles during spreading were almost an order of magnitude smaller than that of the fully molten particles. The pyrometric signals, characteristic of the impact of partially molten zirconia, provide a novel method of identifying partially molten ceramics after impact on a flat surface.

Introduction

In the plasma spraying process, particles are heated and accelerated by a high-temperature ionized gas jet. If the particles are completely molten, they spread, cool, and solidify on the substrate or on previously deposited layers after impact. The quality and mechanical strength of the final coating depends on the morphology and degree of splashing that occurs after impact of the droplets. Pershin *et al.* [1] used plasma-sprayed nickel particles on stainless steel to show that splashing reduces the mechanical strength of a coating due to the increase of porosity and decrease of splat adhesion to the substrate.

In some cases, the impact of partially molten particles will contribute to reducing the overall quality of the coating. Zhang, *et al.* [2] showed that unmelted or partially melted zirconia particles exhibited a significantly different splat

morphology compared to fully molten particles. It was observed that all the partially molten particles fragmented after impact on the substrate. A group parameter, the melting index, was derived to correlate the melting status of the in-flight particles. Lower melting indices indicated that the particles were not fully molten. It was found that if the melting index was lower than 0.2 and the Reynolds number of the in-flight particle was larger than 800, then unmelted fragmentation would occur. Lima and Marple [3] have observed that the presence of nanostructured unmelted particles in a coating improves the coating quality. They found that nanostructured zones embedded in a nanostructured titania coating improved the coating wear resistance by acting as crack arresters.

Cedelle, *et al.* [4] used a fast CCD camera to photograph the different fragmentation phenomena of plasma sprayed yttria partially stabilized zirconia. The CCD camera was oriented either parallel to the cold substrate to photograph the impact splashing or orthogonal to photograph the splashing during flattening. It was found that splashing seemed to occur immediately after impact and continued during flattening. Bianchi, *et al.* [5] showed that the splashing of zirconia was more pronounced on a substrate that was either non-heated or heated to form an oxide layer on the surface, compared to a surface that was heated for a short time period.

In this study, the impact and disintegration of partially molten yttria-stabilized zirconia are investigated. Images of the particles are captured a few microseconds after impact by using a rapid CCD camera and a long-range microscope. A high speed, two-color pyrometer was also used to obtain the temperature evolution during spreading. The two-color pyrometric method, as described by Gougeon, *et al.* [6], was used to calculate the splat temperature and area from the intensities of radiation collected at two different wavelengths. The shape and magnitude of the pyrometric signals were used to identify partially molten particles.

Experimental Method

The experimental assembly and method have been described in detail by Mehdi-zadeh, *et al.* [7]. A schematic diagram of the experimental setup is shown in Figure 1. A SG100 torch (Praxair Surface Technologies, Indianapolis, IN) was used to melt and accelerate yttria-stabilized zirconia (Amperit #825, H. C. Starck, Germany) powder particles, seived to +38 –55 μm . The average diameter of the particles was 40 μm . The powder feed rate was less than 1 g/min. The substrate was a glass microscope slide (Fisher Scientific, Pittsburgh, PA) that was washed with water and ethanol and dried in an oven at 140°C for 30 minutes.

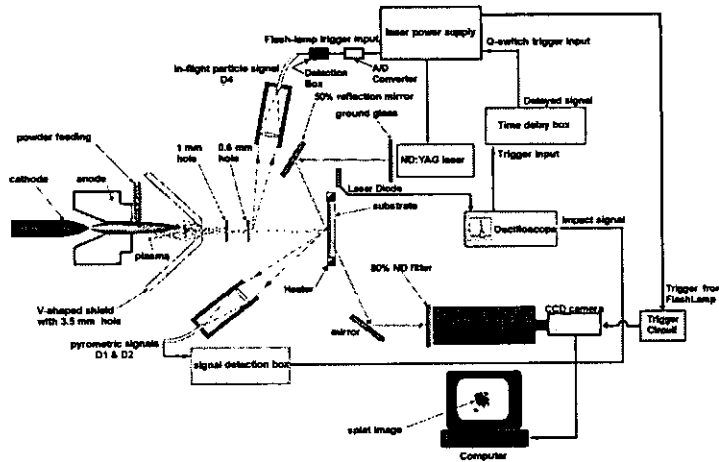


Figure 1: Schematic of the experimental assembly

The plasma torch was passed rapidly across the glass substrate. In order to protect the substrate from an excess of particles and heat, a V-shaped barrier was placed in front of the torch.

The thermal radiation of the particle was measured with a rapid two-color pyrometric system. This system included an optical sensor head that consisted of a custom-made lens, which focused the collected radiation, with a 0.21 magnification, on an optical fiber with an 800 μm core [7]. This optical fiber was covered with an optical mask that was opaque to near infrared radiation, except for three slits. The two smaller slits, with dimensions of 30 μm by 150 μm and 30 μm by 300 μm , were used to detect the thermal radiation of the particles in flight. The radiation was used to calculate the temperature, velocity, and diameter of the in-flight particle [8]. The largest slit, measuring 150 μm by 300 μm , was used to collect thermal radiation of the particle as it impacted and spread on the substrate. With the thermal radiation from this slit, the splat temperature, diameter, and cooling rate were calculated at 100 ns intervals after impact.

The collected thermal radiation was transmitted through the optical fiber to a detection unit that contained optical filters

and two photodetectors. The radiation beam was divided into two equal parts by a beam splitter. Each signal was transmitted through a bandpass filter with wavelength of either 785 nm or 995 nm and then detected using an avalanche silicon photodetector.

The thermal emission signals captured by the photodetectors showed the temporal positions of the particle as it passed through the field of view of each of the optical slits. When the particle was not in an optical field of view, the signal voltage was zero. Two small peaks were produced by thermal emissions from the particle as it passed through the first two small slits. The average in-flight velocity of the droplet was calculated by dividing the known distance between the centers of the two fields of view by the measured time of flight. A plateau on the thermal emission signal corresponded to the presence of the in-flight particle in the field of view of the third and largest optical slit before impact. Upon impact, the signal increased as the particle spread and eventually decreased as the particle cooled down and/or splashed out of the field of view.

To illuminate the impacting particle, a 5 ± 2 ns duration pulse of light from a Nd:YAG laser (Continuum Minilite, Santa Clara, CA) was used. Since the flashlamp of the laser had to be triggered at least 150 μs before it was pulsed, an optical sensor (labelled D₄ in Fig. 1) was positioned to detect thermal radiation from a particle immediately after it exited the 0.6 mm hole in the third shielding plate (Fig. 1). When the pyrometric signal, D₁, exceeded a certain threshold voltage after the particle impact, a signal was sent to trigger the laser after a controlled time delay. This permitted illumination of the substrate at different time intervals after impact and during spreading of the droplet.

A 12-bit CCD camera (QImaging, Burnaby, BC) was used to capture images of the spreading particles. The electronic shutter of the camera was triggered to open by a signal from the flashlamp of the laser. The camera was connected to a long-range microscope (Astro-optics Division, Montpelier, MD) that had an 80% neutral density (ND) filter to attenuate the intensity of the laser beam. The images captured by the camera were digitized by a frame grabber and recorded on a personal computer. Since the images were not photographed directly, but rather, their reflection in a mirror that was at an angle relative to the substrate, the digitized images were rotated and shortened on both dimensions.

Results and Discussion

Impact on Glass at Room Temperature

Thermal emission signals from the D₁ (785 nm) sensor and images of the particles at different times after impact on glass held at room temperature are shown in figure 2. Figure 2a shows the impact of partially molten droplets. The time, $t = 0$ μs , corresponds to the moment of impact. The average in-flight temperature (T_{if}) of the particles was 2520 ± 30 °C,

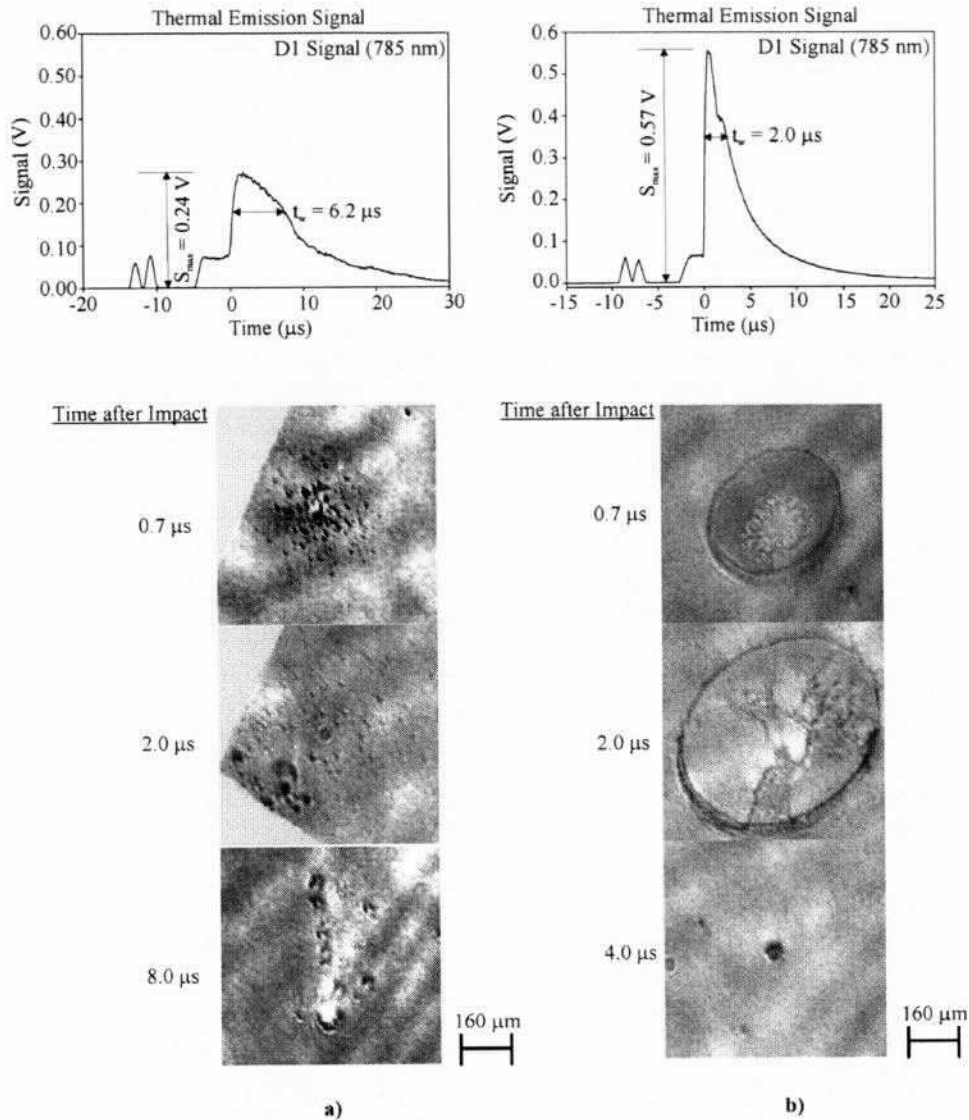


Figure 2: Typical photodetector signals and images of a) partially molten and b) fully molten yttria-stabilized zirconia particles after impact on glass held at room temperature

which is lower than the melting point (2700°C) of yttria-stabilized zirconia. The statistical error of the in-flight temperature is also shown with the average. The statistical errors will be reported with the averages of all other parameters mentioned in this study. These particles disintegrated immediately upon impact into small irregularly shaped fragments (Fig. 2). Figure 2b shows that the fully molten particles, with an average in-flight temperature of $2770 \pm 20^{\circ}\text{C}$, impacted the substrate and spread into a thin liquid film that eventually disintegrated, leaving a solidified central core, $4\text{ }\mu\text{s}$ after impact. The average maximum voltage (S_{max}) of the thermal emission signals of the partially molten particles ($0.24 \pm 0.02\text{ V}$) was approximately 2.5 times smaller than that of the fully molten particles ($0.57 \pm 0.10\text{ V}$).

The total surface area of the fragments of the partially molten particles exposed to the thermal sensors (D_1 and D_2) was lower than that of the molten particles that spread fully into a thin liquid film. Since the thermal emission signal voltage is an indication of the exposed surface area of the splat [8], the signal voltages of the partially molten particles are expected to be lower than the voltages of the fully molten particles that spread completely on the substrate.

The width (t_w) of the thermal emission signal of the partially molten particles were characteristically larger than that of the fully molten particles. The width of the signal was measured at

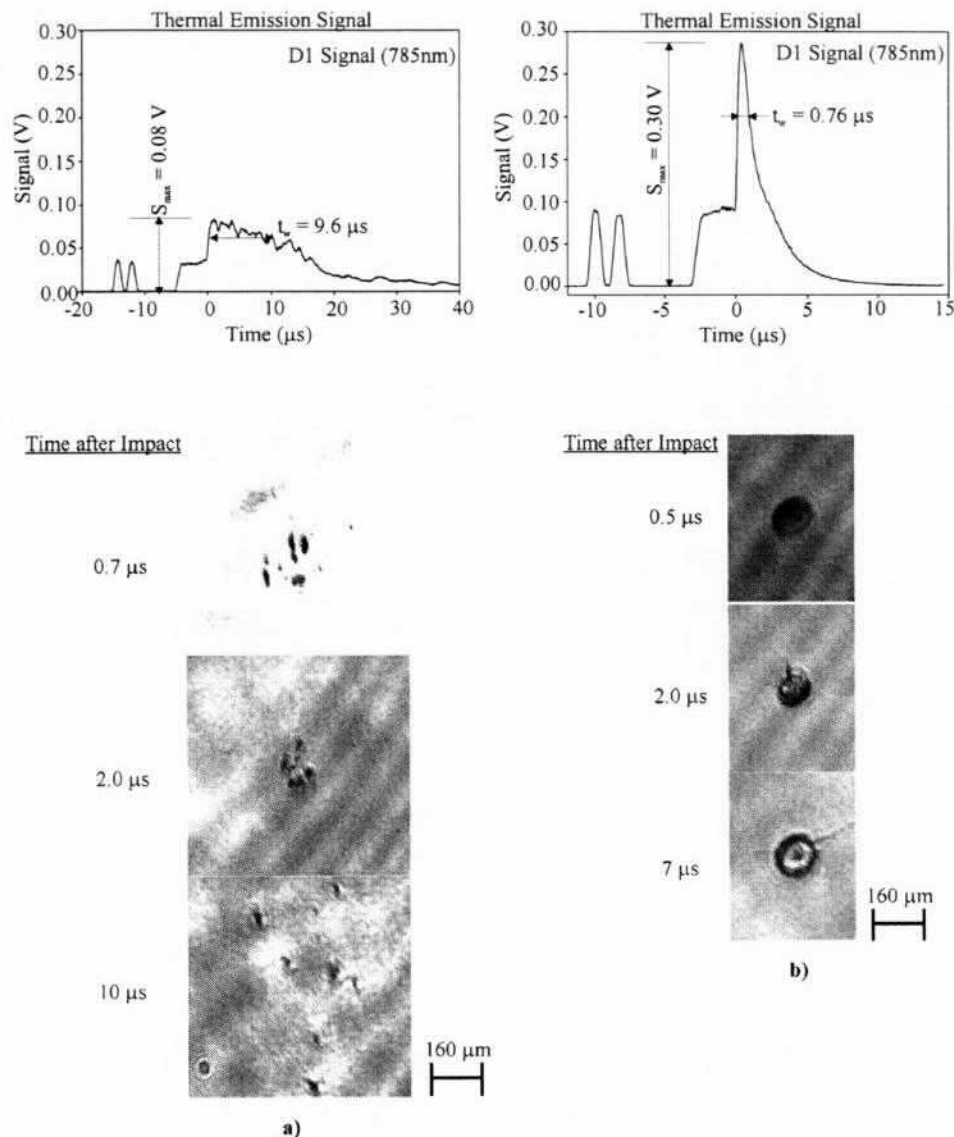


Figure 3: Typical photodetector signals and images of a) partially molten and b) fully molten yttria-stabilized zirconia particles after impact on glass held at 400°C

the half-way point between the plateau and the maximum point. Figure 2 shows that for the partially molten particle, the average width of the signal was approximately $6.2 \pm 0.99 \mu\text{s}$ and for the fully molten particle, it was $2.0 \pm 0.16 \mu\text{s}$.

Impact on Glass held at 400 °C

Figure 3 shows thermal emission signals from the D₁ sensor and images of the particles at different times after impact on glass substrates held at 400 °C. The average in-flight temperature of the partially molten particles of figure 3a was $2460 \pm 30 \text{ }^{\circ}\text{C}$. The partially molten particles also fragment upon impact on the surface held at 400 °C. However, unlike impact on cold glass, more of the fragments on the heated surface solidify and adhere to the surface. McDonald, *et al.* [9]

have shown that the cooling rate of plasma-sprayed yttria-stabilized zirconia on heated glass ($1.9 \times 10^8 \text{ K/s}$) is almost twice as large as the rate on glass held at room temperature ($9.7 \times 10^7 \text{ K/s}$). Since the cooling rate on the heated glass is larger, more of the liquid portion of the partially molten particle solidify and adhere to the substrate (Fig. 5). Figure 3b shows images of the splats of fully molten particles after impact on the heated glass. The average in-flight temperature of these particles was $2730 \pm 20 \text{ }^{\circ}\text{C}$.

The shape of the thermal emission signal of the partially molten particle after impact on the heated glass is similar to that of the particle that impacted glass at room temperature (Fig. 2a and 3a). The average width of the thermal emission

signal of figure 3a is approximately $9.6 \pm 1.7 \mu\text{s}$. For the fully molten particles that impacted and spread on the heated glass, the average width of the signal was $0.76 \pm 0.10 \mu\text{s}$. The maximum voltage of the thermal emission signal of the partially molten particle on this surface ($0.08 \pm 0.01 \text{ V}$) was approximately 4 times smaller than that of the fully molten particle ($0.30 \pm 0.05 \text{ V}$).

Table 1 shows a summary of the average maximum voltages (S_{max}) and widths of the thermal emission signals (t_w) and the average in-flight temperatures (T_{if}) of the partially and fully molten particles that impacted non-heated and heated glass. The average maximum voltages of the particles that impact the cold glass are between 2 to 3 times larger than those of the particles that impacted hot glass. In the case of the fully molten particles, the splats on the cold glass have a larger maximum spread diameter than those on heated glass. The thermal radiation emitted from the larger area will produce a larger maximum voltage [6]. Figure 2a shows that on the cold glass, the hot fragments of partially molten zirconia ($T_{\text{if}} = 2520^\circ\text{C}$) are displaced over a larger area than those fragments ($T_{\text{if}} = 2460^\circ\text{C}$) on heated glass (Fig. 3a). This, coupled with a lower cooling rate, will produce a larger maximum voltage on the thermal emission signal of the partially molten particle on non-heated glass.

Table 1: Average values of the maximum signal voltages, signal widths, and in-flight temperatures of plasma-sprayed partially and fully molten yttria-stabilized zirconia

| | S_{max} | t_w | T_{if} |
|------------------|------------------|-----------------|------------------|
| On cold glass | V | μs | $^\circ\text{C}$ |
| Partially molten | 0.24 ± 0.02 | 6.2 ± 0.99 | 2520 ± 30 |
| Fully molten | 0.57 ± 0.10 | 2.0 ± 0.16 | 2770 ± 20 |
| On hot glass | | | |
| Partially molten | 0.08 ± 0.01 | 9.6 ± 1.7 | 2460 ± 30 |
| Fully molten | 0.31 ± 0.05 | 0.76 ± 0.10 | 2730 ± 20 |

The fully molten zirconia particles that impacted the non-heated glass reached the maximum spread diameter of approximately $450 \mu\text{m}$ after about $2 \mu\text{s}$ after impact. McDonald, *et al.* [10] has shown that the impact of molybdenum, an opaque material, on cold glass produced splats with maximum diameters of $\sim 500 \mu\text{m}$, about $2 \mu\text{s}$ after impact. The maximum voltage of the thermal emission signal obtained by McDonald, *et al.* [10] for molybdenum was $\sim 2 \text{ V}$, while table 1 shows that for fully molten zirconia on non-heated glass, the maximum voltage is 0.57 V . This difference in the maximum voltage was due to the partial transparency of zirconia. Similar observations were made for the impact and spreading on heated glass.

In this study, about 30% of the images and thermal emission signals collected represented particles with in-flight temperatures lower than the melting point of yttria-stabilized

zirconia and had fragmentation morphologies shown in figures 2a and 3a.

After Complete Solidification of the Partially Molten Particles

Figure 4 shows that after impact and disintegration of the partially molten particle on cold glass, only a solidified central core remains. The average diameter of the core is $80 \mu\text{m}$, compared to $75 \mu\text{m}$ when the particle is completely molten (Fig 2b). The formation of the central core is due to the large impact pressures that improve contact between the particle and substrate in that region [7]. According to Li [11], the average diameter of the central solidified core should be 1.5 times the initial, in-flight particle diameter. In this study, the average diameter of the solidified core after impact on glass at room temperature is about 1.8 times the initial particle diameter.

Figure 5 shows the solidified remnants of the original, partially molten particle on the heated glass. The solidified central core is indicated and it is approximately 1.5 times the original diameter of the in-flight particle, as observed by Li [11]. The smaller solidified fragments that adhered to the surface are also shown. On the heated glass, more solidified fragments are seen, probably due to the improved particle contact and adhesion that is typical of impact and spread on heated surfaces [1,5,9,10]



Figure 4: Solidified core of partially molten yttria-stabilized zirconia particles taken a few minutes after impact on glass at room temperature

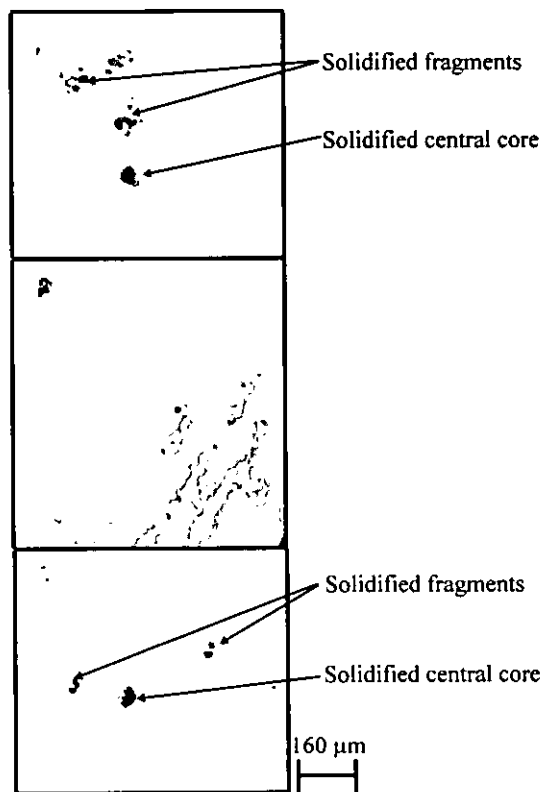


Figure 5: Solidified portions of partially molten yttria-stabilized zirconia particles taken a few minutes after impact on glass at 400 °C

Conclusion

It was shown that the in-flight particle temperature significantly affects the morphology of the particle after impact on a substrate. Yttria-stabilized zirconia particles with in-flight temperatures lower than the melting point disintegrated into small fragments upon impact on glass that was held at either room temperature or 400 °C. The maximum voltages of the thermal emission signals of the partially molten particles, collected after impact by a rapid 2-color pyrometer, was about 2 to 4 times smaller than those of the fully molten particles. This indicated that the exposed surface area of the partially molten particles was lower than those of the fully molten particles. The width of the profiles for the partially molten particles was also characteristically large.

The characteristic shape and size of the thermal emission signals provide an alternate method of identifying partially molten plasma-sprayed yttria-stabilized zirconia particles.

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